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SESSION
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0.21
FULL ESTIMATED COST

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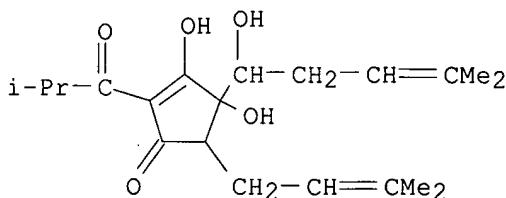
<http://www.cas.org/ONLINE/UG/regprops.html>

=> s 312925-21-6
L1 1 312925-21-6
(312925-21-6/RN)

=> s 24149-26-6
L2 1 24149-26-6
(24149-26-6/RN)

=> d L1 str cn

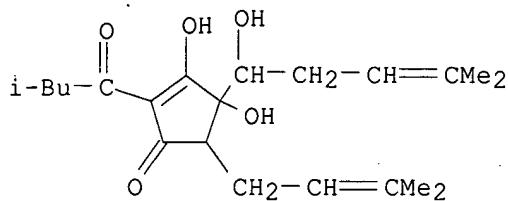
L1 ANSWER 1 OF 1 REGISTRY COPYRIGHT 2006 ACS on STN



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

CN 2-Cyclopenten-1-one, 3,4-dihydroxy-4-(1-hydroxy-4-methyl-3-pentenyl)-5-(3-methyl-2-butenyl)-2-(2-methyl-1-oxopropyl)- (9CI) (CA INDEX NAME)

=> d L2 str cn



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

CN 2-Cyclopenten-1-one, 3,4-dihydroxy-4-(1-hydroxy-4-methyl-3-pentenyl)-5-(3-methyl-2-butenyl)-2-(3-methyl-1-oxobutyl)- (9CI) (CA INDEX NAME)
 OTHER CA INDEX NAMES:
 CN 2-Cyclopenten-1-one, 3,4-dihydroxy-4-(1-hydroxy-4-methyl-3-pentenyl)-2-
 isovaleryl-5-(3-methyl-2-butenyl)- (8CI)

=> file caplus medline biosis embase

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	6.00	6.21

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=> s L1 or L2

L3 6 L1 OR L2

=> dup rem L3

PROCESSING COMPLETED FOR L3

L4 6 DUP REM L3 (0 DUPLICATES REMOVED)

=> d 1-6 ibib abs

L4 ANSWER 1 OF 6 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 2004:368873 CAPLUS

DOCUMENT NUMBER: 140:368677

TITLE: Compositions using hops- and rosemary-derived components, triterpenes, and other compounds for the treatment of pathological conditions associated with inflammatory response

INVENTOR(S): Tripp, Matthew L.; Babish, John G.; Bland, Jeffrey S.; Darland, Gary; Lerman, Robert; Lukaczer, Daniel O.; Liska, Deann J.; Howell, Terrence

PATENT ASSIGNEE(S): Metaproteomics, LLC, USA

SOURCE: PCT Int. Appl., 186 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 6

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004037180	A2	20040506	WO 2003-US33362	20031020
WO 2004037180	A3	20040930		
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
US 2004086580	A1	20040506	US 2003-464410	20030618
US 2004115290	A1	20040617	US 2003-464834	20030618
CA 2503196	AA	20040506	CA 2003-2503196	20031020
AU 2003286549	A1	20040513	AU 2003-286549	20031020
EP 1558271	A2	20050803	EP 2003-777751	20031020
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
JP 2006508182	T2	20060309	JP 2005-501640	20031020
PRIORITY APPLN. INFO.:			US 2002-420383P	P 20021021
			US 2003-450237P	P 20030225
			US 2003-400293	A 20030326
			US 2003-401283	A 20030326
			US 2003-464410	A 20030618
			US 2003-464834	A 20030618
			US 2001-885721	A2 20010620
			WO 2003-US33362	W 20031020

OTHER SOURCE(S): MARPAT 140:368677

AB A natural formulation of compds. for modulating inflammation is disclosed. The formulation would also inhibit expression of COX-2, inhibit synthesis of prostaglandins selectively in target cells, and inhibit inflammatory response selectively in target cells. The compns. contain at least one fraction isolated or derived from hops. Other embodiments disclose combinations of components, including at least one fraction isolated or derived from hops, tryptanthrin and conjugates thereof, rosemary, an extract or compound derived from rosemary, a triterpene species, or a diterpene lactone or derivs. or conjugates thereof.

L4 ANSWER 2 OF 6 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 2003:241402 CAPLUS
DOCUMENT NUMBER: 139:275867
TITLE: Quality control of beer hopped with reduced isomerized products
AUTHOR(S): Jorge, Katia; Truco, Luiz C.
CORPORATE SOURCE: Instituto de Quimica - Departamento de Bioquimica, Universidad Federal de Rio de Janeiro-, Rio de Janeiro, Brazil
SOURCE: Cerveza y Malta (2002), 39(156), 35-39
CODEN: CEMADD; ISSN: 0300-4481
PUBLISHER: Asociacion Espanola de Tecnicos de Cerveza y Malta
DOCUMENT TYPE: Journal
LANGUAGE: Spanish
AB The traditional method for measuring beer bitterness by UV absorbance is a suitable quality control method for normally hopped beer. After reduction (hydrogenation) and isomerization of α -bitter acids in hop products the specific UV absorptivity of the active components change and reversed-phase HPLC anal. becomes a better method for beer quality control. The spectrophotometric and HPLC methods were compared for

suitability in control of the composition of different com. reduced isomerized hop products added to unhopped wort and partially kettle-hopped beer. The HPLC method was reliable for quality control of reduced isomerized hop products and the traditional UV spectrophotometric method was suitable for quality control of beer, including beer made with the reduced isomerized hop products as long as an adequate factor was used.

REFERENCE COUNT: 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 3 OF 6 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 2001:135507 CAPLUS
DOCUMENT NUMBER: 134:309895
TITLE: High-performance separation of unmodified and reduced hop and beer bitter compounds by a single high-performance liquid chromatographic method
AUTHOR(S): Harms, Diedrich; Nitzsche, Frank
CORPORATE SOURCE: Konig Brauerei GmbH and Co. KG, Duisburg, D-47138, Germany
SOURCE: Journal of the American Society of Brewing Chemists (2001), 59(1), 28-31
CODEN: JSBCD3; ISSN: 0361-0470
PUBLISHER: American Society of Brewing Chemists, Inc.
DOCUMENT TYPE: Journal
LANGUAGE: English

AB An improved high-performance liquid chromatog. method for the separation of the isoxanthohumol iso- α -acids, rho-iso- α -acids, tetrahydro-iso- α -acids, α -acids, and β -acids in beer is presented. This is the first time a complete separation of all six isomers of iso- α -acids has been described. It is achieved by using an isocratic elution system in beer. A gradient elution method is used for measuring all the main hop-bittering substances such as α -acids, β -acids, iso- α -acids, and all reduced compds. in light-stable beer, hop exts., and wort. A chemical cross-linked "base deactivated" C18 column with a citrate-buffered (pH 7) acetonitrile-methanol gradient mobile phase enables the separation of the beer bitter components within 50 min. The anal. of wort samples was achieved with a tris-buffered (tris(hydroxymethyl)-aminomethane) (pH 7.5) acetonitrile-methanol gradient mobile phase within 60 min. Sample preparation was by preconcn. on SPE C8 cartridges, which gave good reproducibility and separation of bitter components from the beer matrix. Due to the complex matrix of beer and wort, an optimized sample preparation procedure is used to increase the life of the column.

REFERENCE COUNT: 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 4 OF 6 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 2000:869005 CAPLUS
DOCUMENT NUMBER: 134:265506
TITLE: Quality control of beer hopped with reduced isomerized products
AUTHOR(S): Jorge, Katia; Trugo, L. G.
CORPORATE SOURCE: BrewTech, Rio de Janeiro, Brazil
SOURCE: Technical Quarterly - Master Brewers Association of the Americas (2000), 37(2), 219-224
CODEN: TQMBAC; ISSN: 0542-9811
PUBLISHER: Master Brewers Association of the Americas
DOCUMENT TYPE: Journal
LANGUAGE: English

AB The traditional method for measuring bitterness in beer by UV absorbance (ASBC 8th Edition) remains a routine quality control method for normally hopped beer. However, after reduction of isoalpha acids, the specific absorptivity of active components change, and this method is not applicable. This work was intended to adapt math. the spectrophotometric method and to evaluate an HPLC method to control the composition of different

com. reduced isomerized products added to unhopped wort and partially kettle hopped beer. The results obtained show that the HPLC method is reliable for quality control of reduced isomerized products, however it has been demonstrated in this work that by using adequate factors, the spectrophotometric method is also viable for quality control of beer bitterness.

REFERENCE COUNT: 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 5 OF 6 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 2000:652555 CAPLUS
DOCUMENT NUMBER: 134:41242
TITLE: New ways to determine bitter substances in beer and wort
AUTHOR(S): Harms, D.; Nitzsche, F.
CORPORATE SOURCE: Konig, Brauerei GmbH und Co. KG, Duisburg, D-47138, Germany
SOURCE: Monatsschrift fuer Brauwissenschaft (2000), 53(7/8), 143-146
CODEN: MOBRDJ; ISSN: 0723-1520
PUBLISHER: Fachverlag Hans Carl
DOCUMENT TYPE: Journal
LANGUAGE: German

AB The simultaneous HPLC determination of iso- α acids, tetrahydro-iso- α acids, α -acids and β -acids is a valuable and reliable tool for the anal. of substances contained in beer. The content of unmodified and reduced bitter hop substances can be determined by means of just one single anal. The used base-activated, cross-linked C18 separating column with a citrate-buffered (pH 7) acetonitril methanol gradients as mobile phase makes complete separation possible within 60 min. An addnl. developed isocratic method to determine iso- α -acids and isoxanthohumol in beer samples permits working at more favorable costs and easy handling. The specimens were prepared with a C8 fixed phase cartridge (SPE). Various beers and hop exts. could be successfully analyzed by this method. This method makes identification of various beer and hop characteristics in respect of their acids and the corresponding isomers possible.

REFERENCE COUNT: 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 6 OF 6 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1969:523657 CAPLUS
DOCUMENT NUMBER: 71:123657
TITLE: Chemistry of hop constituents. XXXV. Interconversion of humulone and isohumulone
AUTHOR(S): Connell, Brian E.
CORPORATE SOURCE: Brew. Ind. Res. Found., Nutfield, UK
SOURCE: Journal of the Institute of Brewing (1969), 75(4), 364-6
CODEN: JINBAL; ISSN: 0046-9750
DOCUMENT TYPE: Journal
LANGUAGE: English

AB Isohumulone reverts partially to humulone in solns. as acidic as pH 2.5, in basic solns., and in sealed tubes heated at 100° for 2 hrs.; 5 hrs. heating produces 10 and 20 hrs., 15% conversion. The reverse reaction does not occur under the same conditions in the same time. After heating humulone 50 hrs. at 100°, 10% isohumulone was produced. Thus, isohumulones cannot be freed of humulone by distillation. Interconversion also occurs between the A and B forms of isohumulone but is believed to take place via humulone as intermediate, not directly.

=> s humulone or humulon
L5 926 HUMULONE OR HUMULON

=> s reduced or hydrogenated
L6 3237279 REDUCED OR HYDROGENATED

=> s L5 and L6
L7 76 L5 AND L6

=> dup rem L7
PROCESSING COMPLETED FOR L7
L8 71 DUP REM L7 (5 DUPLICATES REMOVED)

=> s isohumulone or isocohumulone or isoahumulone
L9 682 ISOHUMULONE OR ISOCOHUMULONE OR ISOAHUMULONE

=> s L8 and L9
L10 26 L8 AND L9

=> s L10 and (AY<2000 or PY<2000 or PRY<2000)
'2000' NOT A VALID FIELD CODE
'2000' NOT A VALID FIELD CODE
2 FILES SEARCHED...
'2000' NOT A VALID FIELD CODE
L11 19 L10 AND (AY<2000 OR PY<2000 OR PRY<2000)

=> d 1-19 ibib abs

L11 ANSWER 1 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1997:798689 CAPLUS
DOCUMENT NUMBER: 128:34911
TITLE: Procedure for the catalytic hydrogenation of terpenoids with polymer-bound transition metals
INVENTOR(S): Bayer, Ernst; Schumann, Wilhelm; Erber, Sebastian; Stengele, Klaus-Peter
PATENT ASSIGNEE(S): Nigu Chemie GmbH, Germany
SOURCE: Ger. Offen., 4 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 19620171	A1	19971127	DE 1996-19620171	19960520 <--
WO 9744304	A1	19971127	WO 1997-EP2525	19970516 <--
W: AU, CN, CZ, JP, RU, US				
RW: AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
AU 9729567	A1	19971209	AU 1997-29567	19970516 <--
EP 901455	A1	19990317	EP 1997-923923	19970516 <--
R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, NL, PT, IE				
PRIORITY APPLN. INFO.:			DE 1996-19620171	A 19960520 <--
			WO 1997-EP2525	W 19970516 <--

OTHER SOURCE(S): CASREACT 128:34911
AB A procedure for the hydrogenation of terpenes with hydrogen in the presence of a transition metal-catalyst is characterized by homogeneous hydrogenation in the presence of a catalyst composed of the reaction product of a group VIII transition metal and a polymer, e.g. polyvinylpyrrolidone, polyethylenimine or a starch derivative. Thus, the exocyclic double bonds of isohumulone are quant. hydrogenated in EtOH over a catalyst prepared from Li2PdCl4 in MeOH and aqueous polyvinylpyrrolidone in EtOH containing NaBH4.

L11 ANSWER 2 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
 ACCESSION NUMBER: 1992:254404 CAPLUS
 DOCUMENT NUMBER: 116:254404
 TITLE: Factors affecting antibacterial activity of hop
 compounds and their derivatives
 AUTHOR(S): Simpson, W. J.; Smith, A. R. W.
 CORPORATE SOURCE: Brew. Res. Found., Redhill/Surrey, RH1 4HY, UK
 SOURCE: Journal of Applied Bacteriology (1992),
 72(4), 327-34
 CODEN: JABAA4; ISSN: 0021-8847
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 AB The antibacterial effect of weak acids derived from the hop plant (*Humulus lupulus L.*) increased with decreasing pH. Anal. of the MIC of such compds. against *Lactobacillus brevis* IFO 3960 over pH 4-7 suggests that undissociated mols. were mainly responsible for inhibition of bacterial growth. The antibacterial activity of trans-isohumulone was .apprx.20 times greater than that of humulone, 11 times greater than that of colupulone, and 9 times greater than that of trans-humulic acid when the degree of ionization was taken into account. Monovalent cations (K+, Na+, NH4+, Rb+, Li+) stimulated antibacterial activity of trans-isohumulone, but the effect was smaller than that observed with H+. The response to divalent cations varied. Ca2+ had little effect on antibacterial activity, whereas Mg2+ reduced activity. Lipid materials and β -cyclodextrin also antagonized the antibacterial action of trans-isohumulone.

L11 ANSWER 3 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
 ACCESSION NUMBER: 1989:532582 CAPLUS
 DOCUMENT NUMBER: 111:132582
 TITLE: Removal of odor-causing substances from hop flavoring materials
 INVENTOR(S): Todd, Paul, H., Jr.; Guzinski, James A.
 PATENT ASSIGNEE(S): Kalamazoo Holdings, Inc., USA
 SOURCE: Ger. Offen., 12 pp.
 CODEN: GWXXBX
 DOCUMENT TYPE: Patent
 LANGUAGE: German
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3811149	A1	19881013	DE 1988-3811149	19880331 <--
DE 3811149	C2	19960801		
US 4778691	A	19881018	US 1987-34917	19870403 <--
GB 2203166	A1	19881012	GB 1988-7447	19880328 <--
GB 2203166	B2	19901121		
NL 8800830	A	19881101	NL 1988-830	19880331 <--
BE 1001473	A3	19891107	BE 1988-381	19880331 <--
CA 1336172	A1	19950704	CA 1988-563198	19880331 <--
AU 8814162	A1	19881006	AU 1988-14162	19880405 <--
AU 609110	B2	19910426		
US 4956195	A	19900911	US 1988-196145	19880519 <--
US 5041300	A	19910820	US 1990-516769	19900430 <--
PRIORITY APPLN. INFO.:			US 1987-34917	A 19870403 <--
			US 1988-196145	A1 19880519 <--

AB Hop flavoring materials (non)reduced α - and iso- α -acids (e.g. humulones and isohumulones) contain substances which cause undesirable odors to develop in alc. beverages. These substances are removed by extracting the flavoring materials with water at a pH >4. An organic solvent, e.g. EtOH, hexane; and a water-soluble salt such as NaCl may be present. The extracted material may be fractionated and mixed with other flavoring materials and EtOH to provide

flavoring materials for alc. beverages.

L11 ANSWER 4 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1988:527339 CAPLUS
DOCUMENT NUMBER: 109:127339
TITLE: Preparation of anactinic additive for malt beverages by isomerization and reduction of α -acids in hop
INVENTOR(S): Chicoye, Etzer; Fly, Walter H.; Goldstein, Henry
PATENT ASSIGNEE(S): Miller Brewing Co., USA
SOURCE: U.S., 5 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4759941	A	19880726	US 1985-728831	19850430 <--
CA 1311702	A1	19921222	CA 1988-565641	19880502 <--
PRIORITY APPLN. INFO.:			US 1985-728831	19850430 <--

AB A hopping material useful as a kettle additive in preparing anactinic malt beverages is prepared by a method which includes first forming an aqueous suspension of crude hops containing α -acids, cellulosic material, alkali hydroxide, and an alkali borohydride. The suspension, which has pH>10.5, is heated to isomerize and reduce the α -acids initially present in the hops. The pH is then lowered to apprx.2 to convert the reduced iso- α -acids to their free acid forms so that they are adsorbed on cellulosic hop material in the reaction mixture. The cellulosic material which now contains the free isomerized and reduced α -acids is isolated and is useful as an anactinic kettle additive which can be stored for prolonged periods with no decomposition. The above method was applied to hop pellets containing humulone 73.1 and lupulone 46.4 mg/g in a solution containing NaBH4 and NaOH, pH 12. The mixture

was heated at 60-65° with N2 sweep and agitation for 3 h, followed by cooling and pH adjustment to 2 with H2SO4. The product contained no unisomerized humulone or reduced isohumulone, a small amount of humilic acid, and expected amount of lupulone.

L11 ANSWER 5 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1982:404686 CAPLUS
DOCUMENT NUMBER: 97:4686
TITLE: Hop extracts
INVENTOR(S): Goldstein, Henry; Fly, Walter; Ting, Patrick; Chicoye, Etzer
PATENT ASSIGNEE(S): Miller Brewing Co., USA
SOURCE: U.S., 7 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4324810	A	19820413	US 1980-154577	19800529 <--
GB 2100719	A	19830106	GB 1981-20742	19810704 <--
GB 2100719	B2	19850130		
DE 3126490	A1	19830120	DE 1981-3126490	19810704 <--
DE 3126490	C2	19850221		
AU 527526	B2	19830310	AU 1981-72600	19810706 <--
CA 1144095	A1	19830405	CA 1981-381373	19810709 <--
PRIORITY APPLN. INFO.:			US 1980-154577	19800529 <--

AB Hop exts. were obtained by treating a crude CO₂ extract of hops with an aqueous reducing solution followed by heating in order to convert the humulones to reduced isohumulones. The resulting reaction mixture is then acidified to give an aqueous phase and an organic phase which are separated by raising the temperature; the organic phase contains

the isomerized and reduced humulones which can be utilized in the subsequent preparation of beer or alc. Thus, the CO₂ extract of

Yakima Cluster hops containing 46.68% α -acids and 32.97% β -acids was treated with an aqueous solution of KBH₄ and KOH heated to 60°. After heating the mixture to 60-65° for 3 h with stirring, the solution was cooled and acidified with HCl to pH 2.0. Reheating the mixture to 60° with shaking caused liquefaction of the reduced hop extract which was separated from the aqueous phase by centrifugation. After washing

the organic phase 3 times with H₂O at .apprx.60°, a yellow semi-solid material was obtained that was useful as an anactinic brew kettle additive.

L11 ANSWER 6 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1980:20508 CAPLUS

DOCUMENT NUMBER: 92:20508

TITLE: Improvement of the bitter properties of hops. Part 1. Photoreaction of α -acids

AUTHOR(S): Viriot, M. L.; Andre, J. C.; Niclause, M.; Bazard, D.; Moll, M.; Flayeux, R.

CORPORATE SOURCE: Ec. Natl. Super. Ind. Chim., Nancy, Fr.

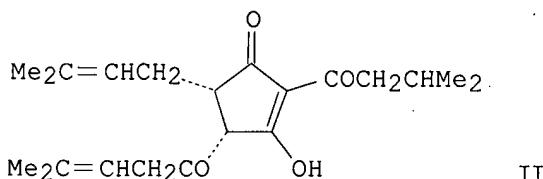
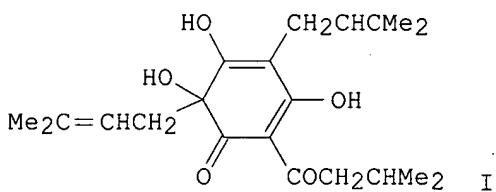
SOURCE: Brauerei-Rundschau (1979), 90(7), 167-9

CODEN: BRRUD8; ISSN: 0379-7716

DOCUMENT TYPE: Journal

LANGUAGE: French

GI



AB Four series of expts. were carried out, investigating the optimum solvent, temperature, pH, and length of time for the isomerization of humulone (I) [26472-41-3] to trans-isohumulone (II) [467-72-1] by UV light. I concns. were .apprx.3 + 10⁻⁴ M. There was little differences among several alc. solvents, but isomerization was slightly better in EtOH and t-BuOH. Isomerization rate increased with temperature from 10° to 63°. Addition of acid showed the isomerization rate, and the isomerized product was destroyed in media. Maximum yield was at .apprx.10 min of UV exposure, after which degradation reduced the yield. Yield was .apprx.90% under the most favorable conditions.

L11 ANSWER 7 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1972:111610 CAPLUS
 DOCUMENT NUMBER: 76:111610
 TITLE: Analysis of hop components by ion exchange chromatography. I. Hops and hop extracts
 AUTHOR(S): Hansen, Gavin L.; Hetzel, Donald; Miller, Robert
 CORPORATE SOURCE: Pfizer, Inc., New York, NY, USA
 SOURCE: Proceedings of the American Society of Brewing Chemists (1971) 246-54
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 AB Ion-exchange chromatog. is used as a method to measure hop acids in both raw hops and hop exts. The procedure, source of reference compds., and calcns. involved are given. A new method of preparing hop samples representing different extraction procedures, and postfermentation exts. containing iso- α - and reduced iso- α -acids, is described. The assay method is reliable, simple to operate, and uses common reagents that are nontoxic and free of peroxide explosion hazards. Sample variables such as age, processing techniques, or solvent residues do not affect the results.

L11 ANSWER 8 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
 ACCESSION NUMBER: 1971:550311 CAPLUS
 DOCUMENT NUMBER: 75:150311
 TITLE: Hop extract
 PATENT ASSIGNEE(S): Pfizer Inc.
 SOURCE: Brit., 3 pp.
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 1246425	-----	19710915	GB 1970-21670	19700505 <--
DE 2106039			DE	
FR 2078451			FR	

PRIORITY APPLN. INFO.: US 19700302 <--
 AB Hops are treated with hexane to extract humulones which are isomerized and reduced by heating in the presence of Na borohydride solution containing NaOH. The reaction solution is acidified and the

reduced isohumulones are extracted with hexane. The improvement consists of converting the reduced isohumulones to H₂O-soluble K salts by mixing the hexane extract with fresh H₂O, heating to 50-60°, adjusting the pH to 7.5-9.0 with aqueous KOH, and separating the aqueous phase containing the K salts of reduced isohumulones. Thus, 212 g of dried ground hops is extracted with 3 l. of refluxing hexane at 64°. The hops are extracted twice more with fresh hexane. The exts. are filtered and concentrated under vacuum to a concentration of 40% solids and 60% hexane. The 72 g of concentrate contains 14.4 g of humulones and 7.2 g of lupulones. To 154 ml H₂O, 9.85 g of 12% Na borohydride solution containing 40% NaOH is added, then heated to 60°. The concentrated hop extract is added and the solution refluxed 3 hr at 60-62° to isomerize and reduce the humulones. The reaction mixture is cooled to 50-60° and acidified to pH 1.6 with an aqueous solution containing 10.3 g H₂SO₄ and 11.1 g (NH₄)₂SO₄. The phases are separated while hot and the aqueous layer is reextd. with fresh hexane at 50-60°, and concentrated under vacuum to yield a hexane solution containing 17.2 g reduced isohumulones and 5 g lupulones. The concentrated solution is mixed with 170 ml H₂O and heated to 50-60°. The pH is adjusted to 7.5-9.0 with 4.65 N KOH. The phases are separated while hot and the hexane phase is re-extracted with 80 ml H₂O at pH 7.5-9.0 and 50-60°. The aqueous layers

are combined, filtered, and concentrated under vacuum. The concentrated solution contains 13.4 g of reduced isohumulone K salts and 0.7 g lupulones.

L11 ANSWER 9 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1969:479770 CAPLUS
DOCUMENT NUMBER: 71:79770
TITLE: Isomerizing and purifying hop extracts
PATENT ASSIGNEE(S): Miller Brewing Co.
SOURCE: Brit., 6 pp.
CODEN: BRXXAA
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 1158370	-----	19690716	GB	<--
DE 1792509	-----		DE	
US 3558326	-----	19710000	US	<--
PRIORITY APPLN. INFO.:			US	19670921 <--
AB	Hop extract is rendered free of wax, oils, and non-acid hop components by adding to a H ₂ O-immiscible solvent solution of hop extract containing the impurities			
hop	an aqueous system substantially alkaline with respect to the pKa of the acid			
	components, thus giving a 2-phase liquid system which is heated to isomerize the humulone to isohumulone. The H ₂ O-immiscible solvent containing the waxes, oils, and non-acidic hop components is separated from the alkaline aqueous system now containing the acidic hop components.			

Fresh H₂O-immiscible solvent is added to the alkaline aqueous system, and the aqueous phase containing the acidic hop components is acidified to a pH substantially acid with respect to the pKa of hop acids, causing the hop acids to enter the H₂O-immiscible solvent phase, which is then separated from the acidic aqueous phase. The hop acids are recovered from the H₂O-immiscible solvent. Thus, a hexane extract of hops was concentrated to prepare 250 lbs. of solids as a 40% solution of hop solids in hexane. To this solution was added 110 U.S. gal. of H₂O containing 65 lb. of a 12% solution of NaBH₄ solution in 40% caustic soda. The result was a 2-phase aqueous-hexane system. The pH of the aqueous phase was 12, which is above the pKa of hop acids and resulted in the extraction of hop acids from the hexane solution into the aqueous phase. The hop acids were isomerized and reduced by the NaBH₄ for 3 hrs. at 140°F. with constant agitation. The dispersed 2-phase system was centrifuged, separating the alkaline aqueous phase containing acid components from the hexane phase containing hop waxes, oils, and nonacidic resins. The aqueous phase was placed in

75 gal. of clean hexane, and 62 lb. of 66% H₂SO₄ was added. The pH of the aqueous phase was 1.5, which is substantially acid with respect to the pKa of hop acids. The 2-phase system was centrifuged, resulting in recovery of 88 gal. of hexane phase, containing 149 lb. of hop solids which analyzed 49% reduced isohumulone, and 125 gal. of aqueous phase. When evaporated to dryness, the hexane phase contained a preisomerized hop extract free of waxes, oils, and nonacidic resins, suitable for emulsifying or solution in an organic solvent for post-kettle addition. The mixture of reduced isohumulones and lupulones is used in brewing.

L11 ANSWER 10 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1968:28487 CAPLUS
DOCUMENT NUMBER: 68:28487
TITLE: Isomerized hop extracts containing
isohumulones
PATENT ASSIGNEE(S): Stafford Allen and Sons, Ltd.
SOURCE: Fr., 3 pp.
CODEN: FRXXAK
DOCUMENT TYPE: Patent
LANGUAGE: French
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 1481292		19670519	FR	19660503 <--

AB The hop flowers (preferably dried and crushed) are treated at 20-5° with a water-immiscible solvent (light petroleum, C₆H₆, CHCl₃, or CC₁₄); the crude solvent extract is contacted with aqueous 2-20% KOH or NaOH to extract humulones. These are isomerized by boiling the alkaline extract (pH 10.7-12) for ≥ 1 hr., optionally under an inert atmospheric (CO₂). The isomerizate is purified by addition of HCl and sufficient MeOH and water to provide a final concentration of 80-5% MeOH. Thus, 45.36 kg. crude C₆H₆ extract was stirred with 47.3 l. aqueous 10% KOH; crude C₆H₆ extract was stirred with 47.3 l. aqueous 10% KOH; after several hrs. the decanted aqueous phase was washed with 18.925 l. C₆H₆. The original C₆H₆ phase was reextd. with 3.785 l. water, and this latter aqueous phase was washed with the 2nd C₆H₆ phase. The combined aqueous exts. were boiled for 1 hr. and then concentrated under reduced pressure to give 45.36 kg. of isomerized product (16% H₂O). This was dissolved in 75.7 l. MeOH; 9.46 l. aqueous 4N HCl and 5.68 l. H₂O were added and the mixture centrifuged. The residue was washed 3 times with 9.46 l. 80% MeOH, the combined MeOH exts. distilled to remove the MeOH, cooled, and mixed with 18.925 l. aqueous 2N KOH; 22.68 kg. dry product was obtained on evaporation

L11 ANSWER 11 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1967:432825 CAPLUS
DOCUMENT NUMBER: 67:32825
TITLE: Borohydride reduction of isohumulones
AUTHOR(S): Khokher, A.; Anteunis, M.; Verzele, Maurice
CORPORATE SOURCE: State Univ., Ghent, Belg.
SOURCE: Bulletin des Societes Chimiques Belges (1967
, 76(1-2), 101-10
CODEN: BSCBAG; ISSN: 0037-9646

DOCUMENT TYPE: Journal
LANGUAGE: English

GI For diagram(s), see printed CA Issue.

AB Isohumulone-A (I) was obtained in 50% yield by irradiation for 20 hrs. of 3 g. humulone in 200 ml. MeOH. I (1.5 g.) in 50 ml. isoctane shaken 18 hrs. at room temperature with 0.35 g. NaBH₄ in 50 ml. H₂O, the mixture acidified to pH 1-2 and extracted with isoctane, and the product (1.41 g. oil) separated by countercurrent distribution gave 3:1 p- isohumulone-A₁ (II), m. 80°, and -A₂ (III), an oil. Isohumulone-B (IV) was obtained by countercurrent distribution of the 2 isohumulones obtained by alkaline treatment of humulone. Reduction of IV by 4 equi. NaBH₄ gave 1:1 p- isohumulone-B₁ (V), m. 78-80°, and -B₂ (VI), an oil. N.M.R., mass, ir, and uv spectroscopy confirmed the structure assigned to II, III, V, and VI. Chemical evidence supporting the structures was also obtained. On boiling in 0.1N NaOH, II and III were not hydrolyzed to humulinic acid, in contrast with the behavior of non-reduced

isohumulones, proving the isohexenoyl side chain is involved in the reaction. II (1 g.) was treated with 1.2 equivs. 0.2M NaIO₄ solution in 50 ml. dioxane under N and the mixture kept 48 hrs. at room temperature; 1 mole NaIO₄ was consumed; the product was dehydroisohumulnic acid (VII), yellow, m. 134°. The structure of VII was determined by mass and uv spectroscopy, and by its identity with the product obtained by bismuth oxide oxidation of humulnic acids. Similarly, on periodate treatment for 4 hrs., 500 mg. III gave 300 mg. VII; 100 mg. each of V and VI gave 60 mg. VII (48-hr. and 4-hr. treatments, resp.). In addition to VII, 4-methylpent-3-enal (VIII) was obtained in the periodate cleavage of II. It was identified by retention time in gas chromatog., by a pos. Schiff test, by mass spectroscopy, and by comparison with synthetic VIII. Isohexenoic acid chloride (0.4 mole) added over 1 hr. to a stirred solution of 0.4 mole ethylenimine and 0.4 mole Et₃N in 50 ml. Et₂O cooled in an ice-salt mixture, the mixture stirred an addnl. 30 min., and the solution after filtration cooled to 0°, treated with stirring over 30 min. with 50 ml. 1.25M LiAlH₄ in Et₂O, stirred an addnl. hr., and treated slowly with cold 5N H₂SO₄ gave VIII (contaminated with the α , β -unsatd. aldehyde). The absolute values of the optical activity of II and V and of III and VI (D line) were very similar but more different at $\lambda = 436$ m μ , indicating that the asym. center at C4 contributed only slightly to total optical activity.

L11 ANSWER 12 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
 ACCESSION NUMBER: 1967:114597 CAPLUS
 DOCUMENT NUMBER: 66:114597
 TITLE: Water-soluble salts of hop acids
 PATENT ASSIGNEE(S): Steiner, S. S., Inc.
 SOURCE: Brit., 3 pp. Division of Brit. 1058975
 CODEN: BRXXAA
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 1058976	-----	19670215	GB 1966-22266	19630806 <--
PRIORITY APPLN. INFO.:			US	19620815 <--

AB A dry powder consisting of a mixture of H₂O-soluble Na salts of α -acids (a mixture of humulone, co-humulone, and adhumulone) present in hops and Na salts of isohumulone was prepared which is useful as an additive in the brewing of beer. Dried hops (1000 g.; 7.41% α -acids and 5.53% β -acids (principally lupulone) by A.S.B.C. assay) were ground to a powder, extracted with hexane (I) by stirring several hrs., and filtered. This process was repeated twice and the combined I exts. concentrated in vacuo to 3 l. which contained 5.5% solids (166.5 g.; 45.6% α -acids and 29.17% β -acids). To 180 ml. of this solution was added 13 ml. N NaOH and 25 ml. H₂O, stirred 10 min. at 25°, separated, the I layer washed with H₂O, and the combined H₂O exts. freeze-dried under high vacuum to give 5.0 g. dry powder (83.4% α -acids). Similar exts. above 50° reduced the recovery of α -acid salts consequential upon isomeric conversion into isohumulone.

L11 ANSWER 13 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
 ACCESSION NUMBER: 1967:54270 CAPLUS
 DOCUMENT NUMBER: 66:54270
 TITLE: Malted beverage not sensitive to light
 INVENTOR(S): Bayne, Peter D.
 PATENT ASSIGNEE(S): Jos. Schlitz Brewing Co.
 SOURCE: Fr., 3 pp.
 CODEN: FRXXAK
 DOCUMENT TYPE: Patent

LANGUAGE: French

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 1448615		19660805	FR	<--
DE 1302694			DE	
GB 1119802			GB	
US 3418135		19681224	US 1964-400580	19640930 <--
PRIORITY APPLN. INFO.:			US	19640930 <--

AB In alkaline solution, humulone (I) in hops extract is converted into isohumulone (II), lupulone (III) is extracted at pH 6-7, then II is extracted at pH >3 and reduced by Na₂S₂O₄. The product, added to a malted, fermented liquor, is not decomposed by light. Thus 40 g. hops containing 2.482 g. I and 1.312 g. III was extracted by Et₂O. The viscous oil

so obtained was refluxed in 500 cc. 0.01N K₂CO₃ solution for 30 min., cooled, and extracted by petroleum ether first at pH 6.5, then at pH 2. The last extract

was dissolved in 150 cc. 95% EtOH and contained 1.60 g. II. The reduction of 0.8 g. II in 15 cc. Et₂O by 0.77 g. Na₂S₂O₄ gave 0.57 g., a yield of 73%. Beer containing 12.5 ppm. II was exposed to sunlight in clear glass bottles for 1 hr. The sample containing original II gave the acrid odor of CH₃C(CH₃)₂-CHSH, the sample containing reduced II was free of this odor.

L11 ANSWER 14 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1965:69712 CAPLUS

DOCUMENT NUMBER: 62:69712

ORIGINAL REFERENCE NO.: 62:12409a-d

TITLE: Hop resins and beer flavor. IV. Observations concerning hard resin

AUTHOR(S): Ashurst, P. R.; Whitear, A. L.

CORPORATE SOURCE: Brewing Ind. Res. Found., Nutfield, UK

SOURCE: Journal of the Institute of Brewing (1965), 71(1), 46-51

CODEN: JINBAL; ISSN: 0046-9750

DOCUMENT TYPE: Journal

LANGUAGE: English

AB cf. CA 60, 12631h. Estimation of α -acids by isolation using thin-layer chromatography agreed with the amts. expected from initial analyses using polarimetry. The formation of oxidized products was studied during the bubbling of O₂ through solns. of humulone, colupulone, and isohumulone. O₂ bubbled at the rate of 10 ml./min. through a solution of humulone (3.0 g.) in light petroleum (100 ml.), gave a precipitate after 3 days of a light yellow solid (1.5 g.), m. 83-87°, C 58.4, H 7.4%, with absolute maximum at 261 m μ , E1%1cm. 232 in MeOH, and absolute maximum 270 m μ , E1%1cm. 272 in acidified MeOH. The oxidation product was hydrogenated with Adams' catalyst in MeOH; 1 mole of H was taken up by 200 mg. The hydrogenated product was a yellow solid, m. 70-76°, C 60.7, H 8.0%, absolute maximum in MeOH at 270 m μ , E1%1cm. 210. Colupulone treated similarly with O₂ gave a petroleum-insol. product in 6 days, m. 72-77°, C 60.1, H 7.6%, absolute maximum in MeOH at 275 m μ , E1%1cm. 183; in alkaline MeOH absolute maximum 270 m μ , E1%1cm. 255. Hydrogenation with Adams' catalyst gave a product, m. 60-65°, absolute maximum in MeOH at 273 m μ , E1%1cm. 187. Oxidation of isohumulone A gave after 5 days an insol. product b0.005 130°, C 64.2, H 7.9%, absolute maximum in MeOH at 278 m μ , E1%1cm. 299; in alkaline MeOH absolute maximum at 278 m μ , E1%1cm. 354. Hydrogenation in AcOH gave C 63.6, H 8.1%.

L11 ANSWER 15 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1962:425462 CAPLUS

DOCUMENT NUMBER: 57:25462
 ORIGINAL REFERENCE NO.: 57:5130d-f
 TITLE: Bittering agents for the production of light-stable malt beverages
 PATENT ASSIGNEE(S): Miller Brewing Co.
 SOURCE: 8 pp.
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 896567		19620516	GB 1960-4403	19600208 <<
US 3044879		19620717	US 1959-792521	19590211 <<
PRIORITY APPLN. INFO.:			US	19590211 <<

AB Reduced isohumulones (I) are used as bittering agents for the preparation of light-stable beer. Thus, hop humulones extracted with petr. ether are evapd, in vacuo. The oily residue is dissolved in MeOH, chilled, and the precipitate filtered off. The filtrate is heated to 60° and 10% methanolic Pb acetate added until complete precipitation. The Pb humulate is digested for 30 min. at 60°, cooled to room temperature, centrifuged, and to the sediment (3 times with MeOH), M H₂SO₄ is added with agitation, and the PbSO₄ precipitate centrifuged off. An equal volume of

2%

H₂SO₄ is added to the supernatant. Humulones are extracted with pert. ether, evapd, in vacuo, dissolved in EtOH, neutralized with 1N NaOH, adjusted to pH 11.0 with 0.2M phosphate buffer, diluted to 1% humulones with water (5 and 10% may be used), and isomerized under reflux for 45 min. This is cooled to room temperature, and the I are reduced by stirring with 700 mg. of KBH₄. (Isomerization and reduction may be done simultaneously.) Here lupulones may be removed by acidifying with 5N HCl to pH 6.4 and extracting with petr. ether. Excess KBH₄ is decomposed by adjusting to pH 2.0 with 6N HCl. Reduced I are extracted with petr. ether, dried in vacuo, added to unhopped wort, and fermented.

L11 ANSWER 16 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1960:128301 CAPLUS
 DOCUMENT NUMBER: 54:128301
 ORIGINAL REFERENCE NO.: 54:24445g-i,24446a-f
 TITLE: Chemistry of hop constituents. XIV.
 2,4-Diacetyl-3,4-dihydroxy-5-methylcyclopent-2-enone,
 an analog of isohumulone A

AUTHOR(S): Brown, P. Margaret; Howard, G. A.

CORPORATE SOURCE: Brewing Ind. Research Foundation, Nutfield, UK
 SOURCE: Journal of the Chemical Society (1960) 164-6

CODEN: JCSOA9; ISSN: 0368-1769

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

OTHER SOURCE(S): CASREACT 54:128301

AB cf. CA 53, 123311. Isohumulone A (I) imparted, from hops to beer, the important bitter taste and bacteriostatic power. Analogs of (I), were not synthesized before. A solution of 12.5 g. dimethylphloroacetophenone (II) and 24 g. Pb(AcO)₂·3H₂O (III) in 400 ml. MeOH was shaken with O. When 500 ml. O had been taken up (4 min.), the rate dropped; 21 g. precipitate was filtered off and decomposed with H₂S in MeOH to

give 5.5 g. product. Upon concentration, the filtrate gave 4.5 g. starting material, m. 22830°. This was treated again with O to give a final product of 2.2 g. and dilution of the filtrate with H₂O gave 2 g. more starting material. The crude yield totaled 7.9 g. m. 156° and was recrystd. from H₂O to give 6.5 g. pure 4-acetyl-5,6-dihydroxy-2,6-dimethylcyclohex-4-ene-1,3-dione (IV), m. 157.5-59°, with λ 230, 240, 283, and 315 m μ (ϵ 10,800, 10,200, 9650, and 6400,

resp.) in acid-MeOH and 257 and 310 m μ (ϵ 19,400 and 12,800, resp.) in alkaline-EtOH. AgNO₃NH₄OH was not reduced, but Folin-Denis reagent reacted to give a blue color. Another analog was prepared by dissolving 2.2 g. IV and 5.2 g. Na₂CO₃ in 100 ml. H₂O and raising to the b.p. in 2 min. After boiling 2 min., the solution was cooled rapidly and acidified at 0°. It was then saturated with NaCl, extracted with EtOAc, the extract washed with brine, and evaporated to give a 2,4-diacetyl-3,4-dihydroxy-5-methylcyclopent-2-enone (V), m. 118-19° (H₂O). The compound sublimed in vacuo, gave a red color with FeCl₃-MeOH, was acidic to litmus, did not reduce Fehling's solution, AgNO₃-NH₄OH, or Folin-Denis reagent, had λ 227 and 265 m μ (ϵ 10,900 and 9100, resp.) in acidic EtOH and 253 and 267 m μ (ϵ 19,200 and 18,800, resp.) in alkaline EtOH. In 0.2M Na-PO₄ buffers, λ 253 and 270 m μ (ϵ 15,200 and 12,200, resp.) at pH 1.0, and 253 and 270 m μ (ϵ 18,800 and 14,900, resp.) at pH 2.4 and 4.4. The anil m. 168° (MeOH), λ 245 and 315 m μ (ϵ 12,700 and 22,000, resp.) in EtOH, and 270 m μ (ϵ 20,300) in alkaline EtOH. The p-chloroanil m. 155° (EtOH); the 2,4-dichloroanil, m. 168° (EtOH). The third analog was prepared by dissolving 500 mg. V in 7.5 ml. EtOH and 25 ml. aqueous N NaOH and refluxing 4 hrs., after which the mixture was poured onto ice. The solution was acidified at 0°, extracted with EtOAc, the exts. washed with brine, dried with MgSO₄ and evaporated to 326 mg. (m. 92-4°) which sublimed at 100°/2 mm. gave 2-acetyl-3,4-dihydroxy-5-methylcyclopent-2-enone (VI) m. 111-12°. The compound gave an orange color with MeOH-FeCl₃. The anil m. 205° (EtOH); the p-chloroanil m. 177° (EtOH); and the 2,4-dichloroanil m. 204° (EtOH). The 4th analog was prepared by dissolving 43 mg. VI in 5 ml. MeOH and titrating conductimetrically with 415 mg. III in 10 ml. MeOH. No break was noted in the curve. After 4 ml. solution was added and left 10 min., 19 mg. orange-brown precipitate was filtered off, suspended in MeOH, and treated with H₂S. PbS was removed and the filtrate evaporated to dryness. The residue was crystallized from H₂O, sublimed at 180°, and recrystd. from H₂O to give yellow 4-acetyl-5-hydroxy-2-methylcyclopent-4-ene-1,3-dione, m. 208-10°; green with MeOH-FeCl₃. The compound showed λ 253 and 275 m μ (ϵ 21,900 and 16,300, resp.) in acid-EtOH and 272 and 300 m μ (ϵ 26,000 and 22,300, resp.) in alkaline-EtOH. The compds. were tested with a beer-infecting Lactobacillus. Inhibition by I was complete at 100 p.p.m. Compds. IV and V were innocuous at this concentration. Infrared spectral data were given for Nujol mulls of dimethylphloroglucinol and II, the analogs of humulone, isohumulone, and humulinic acid, and the anils of V and VI.

L11 ANSWER 17 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN
 ACCESSION NUMBER: 1959:67838 CAPLUS
 DOCUMENT NUMBER: 53:67838
 ORIGINAL REFERENCE NO.: 53:12331i,12332a-i,12333a-b
 TITLE: Chemistry of hop constituents. XIII. Hydrogenation of isohumulone
 AUTHOR(S): Brown, P. Margaret; Howard, G. A.; Tatchell, A. R.
 CORPORATE SOURCE: Brewing Ind. Research Foundation, Nutfield, UK
 SOURCE: Journal of the Chemical Society (1959)
 545-51
 CODEN: JCSOA9; ISSN: 0368-1769
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable
 AB cf. C.A. 52, 12818e. Isohumulone A (I) (853 mg.) was hydrogenated in 70 ml. MeOH over 100 mg. PtO₂; extraction with Et₂O followed by aqueous Na₂CO₃ and distillation at 130° and 2 + 10⁻³ mm. gave neohydroisohumulone (II), λ 253 m μ (ϵ 11,800) and 274 m μ (ϵ 17,200) in acidic and alkaline EtOH, resp. II tasted bitter, gave no copper complex soluble in CHCl₃, failed to reduce Fehling solution, gave a pos. CHI₃ reaction, and was unaffected by boiling N alc. alkali or by KHSO₄ in boiling PhMe. A trace of Me₂CO was produced with

O3. Similar hydrogenation of 2 crystalline isohumulones [m. 123-4°, $[\alpha]D$ -15.7° (MeOH); m. 129-30°, $[\alpha]D$ -40.6° (MeOH)] gave products which failed to distil at 135° and 10-4 mm., but which had ultraviolet light absorption identical to that of II. Hydrogenation of 617 mg. I in 20 ml. HOAc over PtO₂ gave 70% tetrahydroisohumulone (III), m. 31-3°. Hydrogenation of 616 mg. I in 5 ml. MeOH and 50 ml. aqueous 2N Na₂CO₃ over PtO₂ gave dihydroisohumulone A (IV), λ 225 and 274 $\mu\mu$ (E1%1cm. 310 and 256) in acidic EtOH and 252 and 272 (inflection) $\mu\mu$ (E1%1cm. 405 and 350) in alkaline EtOH, iodine value 72, after chromatographic separation from silica gel using C₆H₆. IV gave a brown color with FeCl₃ in MeOH. Hydrolysis with aqueous alkali in EtOH gave γ -methylvaleric acid (V) in low yield. Hydrogenation over Pd-C gave a product with ultraviolet light absorption like that of I, III, and IV. Trituration with C₆H₆ gave a soluble and an insol. fraction in thep roportion 2:1, both of which gave dihydrohumulnic acid (VI) and V after alkaline hydrolysis. Countercurrent distribution of the soluble and insol. fractions gave 71 and 50% III, resp., m. 32-4°, $[\alpha]D$ 24.5° and 98° in neutral and alkaline MeOH, resp., λ 230 and 275 $\mu\mu$ (E1%1cm. 250 and 250) and 253 $\mu\mu$ (E1%1cm. 456) in acidic and alkaline EtOH, resp. Hydrogenation of III in MeOH over PtO₂ gave II. III had a partition coefficient 1.04 in Me₂CHAm-phosphate buffer (0.5M; pH 6.5). Hydrogenation of IV in MeOH over PtO₂ gave II. Reduction over PtO₂ in HOAc gave an oil which distilled at 120° and 5 + 10-4 mm. to give a product with ultraviolet spectrum like that of II but with different infrared absorption and countercurrent distribution. Hydrogenation of 892 mg. isocohumulone A (VII) in 30 ml. HOAc over PtO₂ gave 85% tetrahydroisocohumulone (VIII), m. 40-6°, after chromatography over silica gel with C₆H₆ and distillation at 145° and 10-3 mm., $[\alpha]D$ 28° and 89° in MeOH and alkaline MeOH, resp., partition coefficient 0.47 in the system listed above, λ 230 and 273 $\mu\mu$ (E1%1cm. 294 and 274) and 253 $\mu\mu$ (E1%1cm. 515) in acidic and alkaline EtOH. Tetrahydrocohumulone (IX) (480 mg.) in 74 ml. aqueous 1/15N NaOH was refluxed 9 min. under N. Purification by chromatography gave VIII, m. 55-8° (distilled at 105° and 10-5 mm.), partition coefficient 0.46, $[\alpha]D$ 6° in neutral and alkaline EtOH, λ 230 and 273 $\mu\mu$ (E1%1cm. 264 and 284) and 253 $\mu\mu$ (E1%1cm. 494) in acidic and alkaline EtOH. \pm -IX (2.86 g.) was isomerized in alkaline EtOH to give 1.47 g. VIII, m. 50-2°, λ 230 and 273 $\mu\mu$ (E1%1cm. 260 and 250) and 253 $\mu\mu$ (E1%1cm. 574) in acidic and alkaline EtOH. Refluxing 668 mg. IX with 100 ml. 1/15N NaOH under N 9 min. gave III, m. 49-53°, $[\alpha]D$ -2 and +13° in neutral and alkaline MeOH, partition coefficient 1.04, λ 230 and 275 $\mu\mu$ (E1%1cm. 250 and 265) and 253 $\mu\mu$ (E1%1cm. 480) in acidic and alkaline EtOH. Hydrolysis of 478 mg. III with 7 ml. N NaOH and 3 ml. EtOH at reflux under N 3 hrs. and purification gave V and VI. Oxidation of 200 mg. III in 10 ml. refluxing HOAc with 450 mg. Bi₂O₃ 5 hrs. gave isohumulnic acid, m. 142-3°. III was unchanged by boiling HOAc alone. Similar oxidation of VIII gave isocohumulnic acid, m. 121-3°. Similar oxidation of I gave 5-(3-methylbut-2-enyl)-3-isovalerylcyclopentane-1,2,4-trione. Oxidation of 500 mg. II under reflux in 14 ml. 2N NaOH and 5 ml. 30% H₂O₂ gave 370 mg. of an oil on purification which in turn gave the p-bromophenacyl derivative of V, m. 75-7°. In a similar experiment, 98% the oxidation product was soluble in aqueous NaHCO₃; gas chromatography gave 6:78:17 V, isovaleric acid, and a C₇ acid. After steam distillation of the mixture, the nonvolatile acids were again oxidized with H₂O₂ in boiling alkali; 22% V resulted. Thus, oxidation of II finally gave about 1.9 moles V. Humulone (6.37 g.) was isomerized by Carson's method (C.A. 47, 9926b) to give 729 mg. "isohumulone," m. 124-5°, $[\alpha]D$ 10.4 and 56° in neutral and alkaline MeOH, resp. Countercurrent distribution with Me₂CHAm and phosphate-citrate buffer (pH 5.0) indicated one major component with partition coefficient 1.42, together with minor components. The residue remaining after removal of the "isohumulone" was dissolved in

Et₂O and shaken with 2N NaOH, and the insol. Na salt was removed. The oily isohumulone (3.8 g.) obtained contained 75% I. Hydrogenation of a portion of I over PtO₂ in HOAc gave 60% III. Neohydroisocohumulone (867 mg.) in 20 ml. EtOH was treated overnight with 1.2 g. NaIO₄ in 20 ml. H₂O. After purification the residue was treated with alc. 2,4-dinitrophenylhydrazine-HCl and the hydrazones examined chromatographically on Al₂O₃. No low mol. weight ketones were observed. The alkaline solution after hydrolysis gave V. Infrared spectral data is given

for a

number of the compds. and structures are postulated for II, III, and IV, and the isohumulic acids.

L11 ANSWER 18 OF 19 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1957:94361 CAPLUS

DOCUMENT NUMBER: 51:94361

ORIGINAL REFERENCE NO.: 51:17088e-i

TITLE: Foam properties of beer

AUTHOR(S): Klopper, W. J.

CORPORATE SOURCE: Exptl. Sta. of Dutch Brewing and Malting Ind., Rotterdam, Neth.

SOURCE: Wallerstein Laboratories Communications (1955), 18(No. 61), 123-35

CODEN: WLCOAX; ISSN: 0043-0137

DOCUMENT TYPE: Journal

LANGUAGE: French/Spanish

AB A new procedure was developed for evaluating head retention in beer. Though based on the method of Blom and Prip (C.A. 30, 57201), it provides for characterization of the foam by duration and adhesion, which can be determined in the same test. Foam d. can also be determined and also a Σ (foam stability) value, improved in that it more closely reflects actual foam behavior. Results are given of studies of the reproducibility and "utility" of the determination. The utility number is derived from the ratio of the

over-all range in a series of values to the confidence limit; the higher it is, the more readily the test will detect differences among different lots. Utility values were highest for estimation of foam duration and the improved Σ value; but the adhesion and d. tests were valuable also.

The new procedure was used in a study of the influence of protein-fraction and hop-resin components on foam behavior. The magnitude of the Lundin protein fraction A influences foam duration, but this fraction is not homogeneous and its effect may vary. Thus, when moderate amts. of sugar, rice, maize, or barley were used as adjuncts, the Lundin fraction A in the beer decreased; foam duration increased with barley and was unaffected by the others. Tannin reduced the concentration of Lundin fraction A, and both foam adhesion and foam duration decreased with increasing tannin concentration. Increased use of hops improved both foam duration and foam adhesion but tended to give a bitter flavor. Increased hop boiling time markedly improved foam adhesion but had little effect on foam duration.

Use of humulone or isohumulone in place of hops showed the foam-stabilizing action of hopping as probably largely attributable to isohumulone produced from humulone during hop boiling.

Accordingly, although foam duration appeared to increase with the concentration of high-mol. proteinaceous matter and with the hopping rate, improvement of foam adhesion appeared almost entirely dependent upon increased hopping rate and longer hop boiling time and more specifically on humulone transformation products, probably isohumulone.

L11 ANSWER 19 OF 19 BIOSIS COPYRIGHT (c) 2006 The Thomson Corporation on STN

ACCESSION NUMBER: 1981:174922 BIOSIS

DOCUMENT NUMBER: PREV198171044914; BA71:44914

TITLE: OXIDATIVE DEGRADATION OF ISO HUMULONES IN RELATION TO BEER FLAVOR.

AUTHOR(S): HASHIMOTO N; SHIMAZU T; ESHIMA T

SOURCE: Report of the Research Laboratory of Kirin Brewery Co Ltd,
(1979) No. 22, pp. 1-10.
DOCUMENT TYPE: Article
FILE SEGMENT: BA
LANGUAGE: ENGLISH

AB Isohumulones essentially responsible for the bitterness of beer are also involved in the development of hoppy aroma during wort boiling and of stale (or oxidized) flavor during storage of bottled beer. When hops are boiled in wort, the essential oil of hops is hardly retained in the wort after boiling because of its high volatility and poor solubility in boiling wort. Humulones present in hops are extracted and substantially isomerized to isohumulones. Trace amounts of the isohumulones underwent the oxidative degradation to form the volatile compounds amounting to 300 ppb in wort. Among the volatile compounds, carbonyl compounds including 2-alkanones (C3-C9), alkanals (C2-C10), alkenals (C4-C7), alkadienals (C6-C7) have larger water-solubility than hop oil and survived the vigorous boiling process of wort to give a hoppy aroma. The oxidative degradation of isohumulones took place also in bottled beer during prolonged storage on the shelf and resultant aldehydes essentially caused stale flavor. Because the double bond and the carbonyl group in isohexenoyl side-chain of isohumulone molecule are involved in this degradation, dihydro- (or tetra or hexahydro-) isohumulones of which the side-chain is reduced are more stable to the oxidative degradation. Use of these reduced isohumulones as bittering agents instead of the isohumulones derived from hop-boiling in wort effectively suppressed the development of stale flavor in beer.

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EAST Search History

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L1	9	"1558271"	USPAT	OR	ON	2006/10/11 11:48
L2	22	"1558271"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2006/10/11 12:01
L3	45	"3044879"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2006/10/11 12:06
L4	15	"3418135"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2006/10/11 12:24
L5	13	"1246425"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2006/10/11 12:31
L6	27	"4324810"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2006/10/11 12:36
L7	4	"5041300"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2006/10/11 12:51

EAST Search History

L8	19	"1058976"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2006/10/11 12:52
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S23	1	"20020086070"	US-PGPUB; USPAT	OR	ON	2006/10/11 09:48
S24	3	"9944623"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2006/10/04 14:49
S25	101	Kuhrts.IN.	USPAT	OR	ON	2006/10/05 14:49
S26	101	S25	USPAT	OR	ON	2006/10/05 14:50
S27	101	S25	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2006/10/05 14:52
S28	1	S25 and hops	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2006/10/05 14:50
S29	2	"20040137096"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2006/10/05 14:52
S30	281	"hops extract"	USPAT	OR	ON	2006/10/11 09:48
S31	281	S30	US-PGPUB; USPAT	OR	ON	2006/10/11 09:48
S32	220	humulone or humulon	US-PGPUB; USPAT	OR	ON	2006/10/11 09:49
S33	93	S31 and S32	US-PGPUB; USPAT	OR	ON	2006/10/11 10:21

EAST Search History

S34	12	dihydroisohumulone	US-PGPUB; USPAT	OR	ON	2006/10/11 09:51
S35	0	"2004086580"	US-PGPUB; USPAT	OR	ON	2006/10/11 10:21
S36	0	"2004037180"	US-PGPUB; USPAT	OR	ON	2006/10/11 10:22
S37	11	"2004037180"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2006/10/11 10:27
S38	10	"1481671"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2006/10/11 10:32
S39	17	"677289"	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2006/10/11 10:32